Cairo, January 15, 2020

SAMPLING
Bulk shipments (GRAINS)
Sampling

**Sampling is a procedure which requires both method and equipment that are suitable for the task.** Any analysis of a consignment and any interpretation of the results would be useless if the sample were not representative of the consignment from which it was taken.

**SAMPLING INCLUDES THE FOLLOWING STAGES:**

a) taking a defined number of increments to constitute an aggregate sample representative of consignment sampled;
b) homogenization of the aggregate sample (dividers);
c) reduction of the aggregate sample into laboratory sample(s) for further testing (in ISO17025 accred. laboratory).

**REPRESENTATIVE SAMPLE IS/WHEN:**

- Proper sampling with most suitable sampling equipment;
- Appropriate number of samples and increments taken;
- Points from where samples are taken;
- Mixing of samples;
- Packing, labelling, storage.
Sampling

Representativeness of sampling is highly important.

All standards vote for sampling when the products are flowing (e.g. during loading or unloading) as most reliable method, so that all the constituent parts of the lot have the same probability of being sampled.

However, if the above is not possible, for non-flowing large commodities (static, i.e. cargo in vessel’s holds), particular care shall be taken to ensure that these increments are distributed regularly throughout the grain mass, both at the surface and deep down.

If we talk about sampling of static grain, the increments should be taken at regular intervals over the entire width and depth, up to 9 m. It is preferred to sample lots of bulk grain which are more than 9 m deep when they are flowing (ISO24333/ 5.2.3).
Equipment and devices

is important and integral part of sampling.

There are many different types of sampling equipment or devices.

The most suitable equipment should be chosen considering the product to be sampled, the quantity required and the containers to be used.
Equipment and devices (Manual samplers)

- Ellis cup and Pelican are designed for sampling of cargo in flow;

- Probe samplers (2-3 m in depth) are for static cargo.
Equipment and devices (Automatic samplers)

Automatic sampler from vehicles is programmed for taking fixed quantity of samples/increments from each vehicle (min 3-5 increments).

Automatic sampler for bulk loading, this automatic sampling device forms an integral part with the loading equipment and makes periodic increments basis given settings (depending on loading rate).
Equipment and devices (Vacuum samplers):

Market offers a pneumatic sampling systems up to 60ft. (18m) deep.

As advised by producer, two men can make six or seven 60-foot probes in the span of a working day.

The probe itself is a series of sections of inner and outer tubes. The chamber formed between these two tubes allows air to pass downward to the tip of the probe where the pneumatic action takes place. As the air flow is moving downward it creates a vacuum, “lifting” the sample upward, forcing the probe further into the mass.

Note: due to vacuum tube, such equipment may take in more dust and light fractions than any other mechanical/manual equipment, which may lead to respective differences in loadport vs disport findings of proximate parameters.
Source: youtube.com – SAMPLING by Vac-A-Sample device (published by Majed Abu Zahra)
Turn on the vacuum probe and insert the tube with the tip.

Source: youtube.com – Vacuum probe SAV2000 (published by GehakaOriginal)
SAMPLING METHODS
ISO 24333 (static grain)

ISO24333/5/5.1 The number of increments and the masses for grain are given in Tables 1 (flowing) and 2 (static). One laboratory sample is required by lot or sub-lot of 1500 mt maximum.

EXAMPLE: For a lot of 6 000 mt, analyze at least four laboratory samples.

The number of samples to be taken for laboratory analysis and arbitration shall be subject to an agreement between the parties concerned. (ISO24333/5.2.4.4)

Table 2

<table>
<thead>
<tr>
<th>Size of lot or sub-lot (m)</th>
<th>Range of mass of increment (g)</th>
<th>Minimum number of increments</th>
<th>Minimum mass of laboratory sample for contaminants</th>
<th>Minimum mass of laboratory sample for other analyses</th>
</tr>
</thead>
<tbody>
<tr>
<td>m ≤ 15 t</td>
<td>400 g to 3 000 g</td>
<td>3 sampling points</td>
<td>For ochratoxin A and aflatoxins: 10 kg</td>
<td>1 kg to 3 kg according to the analytical requirements</td>
</tr>
<tr>
<td>15 &lt; m ≤ 30 t</td>
<td></td>
<td>8 sampling points</td>
<td>For pesticides, heavy metals, dioxins: 1 kg</td>
<td></td>
</tr>
<tr>
<td>30 &lt; m ≤ 45 t</td>
<td></td>
<td>11 sampling points</td>
<td></td>
<td></td>
</tr>
<tr>
<td>45 &lt; m ≤ 100 t</td>
<td></td>
<td>15 sampling points</td>
<td></td>
<td></td>
</tr>
<tr>
<td>100 &lt; m ≤ 300 t</td>
<td></td>
<td>18 sampling points</td>
<td></td>
<td></td>
</tr>
<tr>
<td>300 &lt; m ≤ 500 t</td>
<td></td>
<td>20 sampling points</td>
<td></td>
<td></td>
</tr>
<tr>
<td>500 &lt; m ≤ 1 500 t</td>
<td></td>
<td>25 sampling points</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Per lot or sub-lot of 1 500 t</td>
<td></td>
<td>25 sampling points</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

- a) If taken mechanically, the mass of the sample can be appropriate to the equipment.
- b) For grain bulk of great depth, a sample taken every 2 m over a sampling height corresponds to one increment. Repeat the procedure as many times as necessary.
- c) Other contaminants like DON, fumonisins, zearalene; for the determination of DON, the mass of laboratory sample can be 1 kg.

Sampling procedure to obtain the minimum mass of laboratory sample for static grain.

Up to a depth of 2m, manual probes (sampling spear) can be used.

Up to a depth of 2.5m, mechanical sampling devices can be used if the principle of sampling according to which they function does not create any segregation at the probe tube intake and does not cause damage to the grains.

Otherwise, if the height of the product exceeds 2.5 m, only suction (vacuum) sampling devices shall be used. Sample lots of bulk grain which are more than 9 m deep when they are flowing. (Clause 5.2.3)
# ISO 24333 (flowing grain)

**Table 1**

<table>
<thead>
<tr>
<th>Method</th>
<th>Range of mass of increment</th>
<th>Minimum number of increments&lt;sup&gt;a&lt;/sup&gt;</th>
<th>Minimum mass of laboratory sample for contaminants</th>
<th>Minimum mass of laboratory sample for other analyses</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mechanical</td>
<td>300 g to 1 900 g</td>
<td>— 20 per lot or sub-lot of 500 t</td>
<td>For ochratoxin A and aflatoxins: 10 kg</td>
<td>1 kg to 3 kg according to analytical requirements</td>
</tr>
<tr>
<td>Manual</td>
<td>300 g to 1 900 g</td>
<td>— 20 per lot or sub-lot of 500 t</td>
<td>For pesticides, heavy metals, dioxins: 1 kg</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>— 25 per lot or sub-lot of 1 500 t for large batches of size greater than 1 500 t</td>
<td>For other contaminants&lt;sup&gt;b&lt;/sup&gt;: 3 kg</td>
<td></td>
</tr>
<tr>
<td></td>
<td></td>
<td>— 4 per lot or sub-lot of 1 500 t for large batches of size greater than 1 500 t</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

<sup>a</sup> Frequency according to grain flow.

<sup>b</sup> Other contaminants like deoxynivalenol (DON), fumonisins, zearalenone; for the determination of DON, the mass of laboratory sample can be 1 kg.

Sampling procedure to obtain the minimum mass of laboratory sample for **flowing grain**.
GAFTA 124/ 4.4.2 For goods in bulk at discharge

Increment samples shall be drawn uniformly and systematically, concurrently with discharge, from various parts of the hold in a fair proportion, excluding the run. If for any reason they cannot be drawn from the hold, increment samples shall be drawn uniformly and systematically, concurrently with discharge, at the nearest practicable point to the hold, preferably from a moving stream. Increment samples shall be taken by hand scoop or by other mutually agreed equipment throughout discharge.

Table 1: Increment sampling - size of lots, number and size of consignments. (GAFTA 124/ 4.2.2)

<table>
<thead>
<tr>
<th>Consignment size</th>
<th>Tonnes</th>
<th>0-5,000</th>
<th>5,001-10,000</th>
<th>10,001 - 25,000</th>
<th>&gt;25,000</th>
</tr>
</thead>
<tbody>
<tr>
<td>Lot size</td>
<td>Tonnes</td>
<td>500</td>
<td>1,000</td>
<td>2,500</td>
<td>5,000</td>
</tr>
<tr>
<td>No. of increments per lot</td>
<td>Number</td>
<td>min 20</td>
<td>min 30</td>
<td>min 40</td>
<td>min 50</td>
</tr>
<tr>
<td>Min bulk (aggregate) sample per lot</td>
<td>Kilos</td>
<td>20</td>
<td>30</td>
<td>40</td>
<td>50</td>
</tr>
<tr>
<td>Max weight of increments</td>
<td>Kilos</td>
<td>1</td>
<td>1</td>
<td>1</td>
<td>1</td>
</tr>
</tbody>
</table>
Calculation of contractual samples: 6.2 All contractual samples for any purpose shall be not less than 3 kilograms per 5,000 tonnes of contract quantity or part thereof except where such a balance does not exceed 250 tonnes. Unless the contract stipulates otherwise, sets of samples are required as follows:

6.2.1 For Grain […]
One set of samples consists of the following:
Sample 1: for analysis
Sample 2: for arbitration
Sample 3: spare

GAFTA 124/ 6.1.4 Arbitration Samples: Buyers and Sellers are entitled to attend at the point of loading or discharge and jointly seal a set of samples consisting of 3 kilograms per 5,000 tonnes or part thereof for arbitration purposes only.
EU official practice of drawing samples of imported goods:

To the best of our knowledge, official practice by authorities in EU with grains/oilseeds shipments on their arrival for discharge is based on the principle that importers have taken the appropriate steps for product sourcing and have obtained assurances that the imported foods are of “good order” (i.e. that they will meet regulatory requirements).

Generally authorities check if all relevant documents (custom clearance/ phyto/ C.O.O., etc) are present on arrival. Samples might be taken by the authorities (usually during discharge) on random shipments on risk-assessment basis, sampling plan, destination, type of cargo, its purpose, etc. This is based on imported food consignments or lots categorized as: a “high-risk” or a “regular” product.

As a rule, competent authority shall develop a sampling plan (normally with two parts, one for high-risk, and one for regular commodities), based on the risk strategy that identifies the foods to be sampled, what analysis will be carried out, when to sample, where to sample, and actions in the case of a non-compliant result. Under this plan, sampling is undertaken as part of comprehensive regulatory control measures that include assessment of importers’ activities.

The individual results of sampled lots are assessed against regulatory standards and the cumulative data is reviewed on a periodic basis, usually quarterly, in order to determine trends and identify any developing issues and the appropriate response.
COMMISSION REGULATION (EU) No 691/2013 of 19 July 2013 –
methods of sampling and analysis / DEFINITIONS:

— Lot: an identified quantity of feed determined to have common characteristics, such as origin, 
variety, type of packaging, packer, consignor or labelling, and in case of a production process, a unit of 
production from a single plant using uniform production parameters or a number of such units, when 
produced in continuous order and stored together.
— Sampled portion: A lot or an identified part of the lot or sublot.
— Sealed sample: a sample sealed in such a manner as to prevent any access to the sample without breaking or 
removing the seal.
— Incremental sample: A quantity taken from one point in the sampled portion.
— Aggregate sample: An aggregate of incremental samples taken from the same sampled portion.
— Reduced sample: A part of the aggregate sample, obtained from the latter by a process of representative 
reduction.
— Final sample: A part of the reduced sample or of the homogenised aggregate sample.
— Laboratory sample: a sample intended for the laboratory (as received by the laboratory) and can be the final, 
reduced or aggregate sample.
COMMISSION REGULATION (EU) No 691/2013 of 19 July 2013 – methods of sampling and analysis (for feed)

Samples intended for the official control shall be taken according to the methods described below. Samples thus obtained shall be considered as representative of the sampled portions.

Samples shall be taken by persons authorised for that purpose by the competent authority.

The sample has to be sealed in such a manner as to prevent any access to the sample without breaking or removing the seal. Sample has to be indelibly marked and must be identified in such a way that there is an unambiguous link to the sampling report.

From each aggregate sample at least two final samples are taken: at least one for control (enforcement) and one for the feed business operator (defence). Eventually, one final sample may be taken for reference. (Clause 3)
Large lots transported by ship,

The sampling is to be done per hold (entity that can physically be separated).

**Clause 5.3.** The sampling of large lots in ships is preferably carried out while the product is in flow (dynamic sampling). In the case of large sampled portions (sampled portions > 500 tonnes), the number of incremental samples to be taken = 40 incremental samples + $\sqrt{\text{tonnes}}$ in relation to the control of substances or products uniformly distributed throughout the feed or 100 incremental samples + $\sqrt{\text{tonnes}}$ in relation to the control of constituents or substances likely to be distributed non-uniformly in feed materials.

Sampling of lots transported by ship by static sampling.
The sampling has to be performed on the accessible part (from above) of the lot/hold. The number of incremental samples is determined by taking into account the size of the sampled portion (Clause 8.2.2). In the case of large sampled portions (sampled portions > 500 tonnes), the number of incremental samples to be taken = 100 incremental samples + $\sqrt{\text{tonnes}}$ (Clause 5.3.).

Minimum size of aggregate sample (loose feed) 4 kg.
However, the aggregate sample for the control of the presence of genetically modified material must contain at least 35 000 seeds/ grains. This means that for maize the size of the aggregate sample must be at least 10.5 kg and for soybean 7 kg. For other seeds and grains such as barley, millet, oat, rice, rye, wheat and rapeseed, the aggregate sample size of 4 kg corresponds to more than 35 000 seeds. For the control of the presence of pesticide residues in cereal grains, the minimum size of the reduced sample shall be 3 kg. (Clause 6)
Commission Regulations (EC) for border protection for food safety

- For the control of levels of mycotoxins: **Commission Regulation (EC) 401/2006**

- For the control of levels of lead, cadmium, mercury, inorganic tin, 3-MCPD and benzo(a)pyrene in foodstuffs: **Commission Regulation (EC) No 333/2007**

- For the control of levels of dioxins, dioxin-like PCBs and non-dioxin-like PCBs in certain foodstuffs: **Commission Regulation (EU) 2017/644** of 5 April 2017 laying down methods of sampling and analysis for the control of levels of dioxins, dioxin-like PCBs and non-dioxin-like PCBs in certain foodstuffs
Commission Regulations

(EC) No 401/2006 of 23 February 2006 laying down the methods of sampling and analysis for the official control of the levels of mycotoxins in foodstuffs

(EC) No 333/2007 of 28/03/2007 methods of sampling and analysis for the official control of the levels of lead, cadmium, mercury, inorganic tin, 3-MCPD and PAH in foodstuffs

(EU) No 2017/644 of 5/04/2017 methods of sampling and analysis for the control of levels of dioxins, dioxin-like PCBs and non-dioxin-like PCBs in certain foodstuffs

Each lot (shipment) exceeding 1500mt shall be subdivided into 500mt sublots. (Table 1)
Each subplot must be physically separate and identifiable and, if so, sampled separately (at least 10 increments, 100 gram each = 1kg aggregate sample per subplot (Table 3); for mycotoxins (B.3) 100 increments, 100 gram each = 10kg aggregate per subplot).
The weight of an incremental sample shall be at least 100 grams. (B.1)

For mycotoxins (B.3), in case the lot is not or cannot be physically separated into sublots, a minimum of 100 incremental samples is taken from the lot.

Any departure from the method shall be recorded.

If it is not possible to carry out the method of sampling set out in this point because of packaging forms, means of transport, etc., an alternative method of sampling may be applied if it is as representative as possible and is fully described and documented. An alternative method of sampling may also be applied in cases where it is practically impossible to apply the abovementioned method of sampling. This is e.g. the case where large lots of cereals are stored in warehouses or where cereals are stored in silos.
Dividers

If possible and appropriate, apparatus designed to divide the sample into approximately equal parts should be used for the preparation of reduced samples in a representative way.

Coning and quartering method  Divider: multiple-slot divider  Cone-shaped dividers (Boerner type)
Mixing and dividing the samples
CONING AND QUARTERING METHOD

Work on a clean, non-absorbent surface.

First, thoroughly mix the aggregate sample by repeating below operations at least twice:

1. Gather the grains together into a cone-shaped pile.
2. Flatten out the surface of the pile and then divide the pile into quarters, A, B, C, and D.

After that:
1. Discard two diagonally opposed quarters (B and C) and mix the two remaining quarters (A and D) (see Figure 4).
2. Repeat the whole process until the laboratory sample of the required size is obtained.

Figure 4 — Coning and quartering method
Mixing and dividing the samples
DIVIDER: MULTIPLE-SLOT DIVIDER, CONE-SHAPED DIVIDERS

Use an apparatus and collection boxes or buckets, follow the steps below and on the picture:

First, homogenize the sample by repeating below operations at least three times and by remixing the subsamples in the hopper:
1. Pour the aggregate sample into the hopper.
2. Two sub-samples are obtained in the two collection boxes (or buckets). Repeat at least three times.
3. Keep the contents of one of the two collection boxes.
4. Put two empty collection boxes back in position. And continue the above steps.

Repeat operations 1 to 4 as many times as necessary using the sub-sample from 3, alternating the collection boxes to be kept until the laboratory sample of the required size is obtained.

Please see video below for more details.
Samples preparation

Samples should be thoroughly mixed using dividers or manual quartering; well packed in rigid airtight and moisture-tight containers fitted with airtight and moisture-tight closures. The containers shall be completely filled and the closures shall be sealed.
### Samples labelling and storage

**GAFTA124/5**

<table>
<thead>
<tr>
<th>Sender</th>
<th>M.V.</th>
<th>From</th>
<th>To</th>
<th>Commodity</th>
<th>Bags/Bulk</th>
<th>Marks</th>
<th>Shipper/Sellers/Buyers</th>
<th>Set No</th>
<th>Sample No</th>
<th>Date and Place of Sealing</th>
<th>Quantity represented by this sample</th>
<th>Part Total Quantity of</th>
<th>*Arbitration (Quality/Rye Terms), Natural Weight/Analysis</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
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</tr>
</tbody>
</table>

* delete as appropriate

<table>
<thead>
<tr>
<th>D/O</th>
<th>Receiver</th>
<th>Quantity</th>
</tr>
</thead>
<tbody>
<tr>
<td>B/L</td>
<td></td>
<td></td>
</tr>
<tr>
<td>No.</td>
<td></td>
<td></td>
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<tr>
<td>Seals</td>
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<td></td>
</tr>
</tbody>
</table>

**ISO24333/7.2**

- a) the nature of the product;
- b) the mass represented;
- c) the lot identifier;
- d) the contract number (if necessary);
- e) the sampling date;
- f) the location and point of sampling;
- g) the name of the person who carried out the sampling.

**STORAGE:** Contractual/arbitral samples are normally stored for 3 months from BL date (according to GAFTA) or as instructed by Principal.